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X-RAY ANALYSIS OF TITANIUM COATINGS AND FILMS

by

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OBJECTIVE

Determine the crystal orientation of corrosion resistant titanium coatings on mild steel and thin titanium films evaporated on pure iron and mica surfaces.

RESULTS

An x-ray analysis was performed on titanium coatings and films prepared under various conditions. Pole figures and diffraction patterns of various crystal planes were obtained.

CONCLUSIONS

Since the nature of the vacuum vapor diffusion coating process precludes the formation of an oriented crystal structure, a post-heat or mechanical treatment seems required to produce a preferred crystal orientation in the protective overlayer.

ADMINISTRATIVE INFORMATION

This program was supervised and conducted by the Physical Sciences Laboratory of U. S. Army Tank-Automotive Center under DA Project 597-01-006, AMSC Code No. 5016.11.844, CRN 13708-37.

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ABSTRACT

Geiger counter and x-ray film techniques were used to study the crystalline structures of thin titanium films and coatings formed by vacuum deposition. An x-ray analysis of the various titanium films was used to determine the effects of temperature and base metal orientation upon the structures of the deposited films. Results indicate that the degree of preferred orientation of the films can be altered by variations in the vacuum deposition technique. Attempts were made to correlate film orientation with adhesion and corrosion resistance properties.

OBJECTIVE

1. Determine the crystal orientation of titanium coatings on ferrous and non-metallic surfaces using standard Geiger counter and film techniques.

2. Evaluate the corrosion resistance properties of titanium deposited below the diffusion temperature.

SUMMARY

The crystalline orientation of vacuum-deposited titanium coating on steel, a corrosion resistance process developed at the U. S. Army Tank-Automotive Center, has been studied by x-ray pole figure and film techniques. Pole figures of the basal (002) plane of thin titanium films were obtained by the Schulz Transmission technique while diffraction patterns on a thick steel backing were obtained by a low angle reflection technique.

CONCLUSIONS

1. Although mechanically bonded titanium, which is formed by depositing on steel below the diffusion temperature, has good mechanical characteristics, it does not possess reliable corrosion resistant properties.

2. The diffusion-type coating of titanium, which has excellent corrosion resistance, consists of a random crystal orientation. Coatings made below the diffusion temperature have a crystal orientation, which appear to be dependent upon substrate temperature.

PROJECT TITLE: X-RAY ANALYSIS OF TITANIUM COATINGS AND FILMS

INTRODUCTION

Because of its low density, high strength, and excellent corrosion resistant properties, much interest has been shown in the utilization of titanium metal for various structural purposes. Within the past few years, pure titanium and titanium alloys have been extensively used in the fabrication of components that are exposed to highly corrosive environments. By mechanically hot-rolling steel-plate with a titanium sheet, the corrosion resistance of steel has been greatly increased.

Initial work on the use of vacuum deposition of corrosion resistant titanium coatings for the protection of steel against atmospheric and cathodic corrosion was recently reported by Physical Sciences Laboratory, U. S. Army Tank-Automotive Center[1]. This report outlined the development of a vacuum vapor deposition technique in which a diffusion layer of titanium was formed on steel test panels. During the deposition process, the metal was vaporized in a high vacuum, and upon condensing on a surface, the atoms of the vapor formed their characteristic crystal structure on the surface of the base metal. If steel was kept below the diffusion temperature, the titanium formed a non-diffusion or mechanical bonded layer on the surface. However, by heating the steel at or above this temperature, the titanium vapor diffused into the steel and formed a metallurgical bond between the titanium and the base metal.

During the early stages of condensation, the crystal orientation of the deposited layer is noticeably influenced by the crystal orientation of the surface[2]. It has been suggested by Uhlig[3] that there might be a relationship between the type of crystal orientation of the film and corrosion rates. A study of the preferred orientation of thin titanium films from pole figures plotted by x-ray diffraction techniques may provide useful information concerning these two phenomena.

The Physical Sciences Branch of the Army Tank-Automotive Center has conducted several investigations on the vacuum deposition of metals as protective coatings for Ordnance materials. Recently, this Laboratory has published a report on the development of vapor deposited corrosion resistant titanium coatings on mild steel panels. Various factors, vital in the vacuum deposition process, such as deposition technique, metal purity, and surface preparation were thoroughly investigated and evaluated. One of the conclusions of this report was that in order to obtain a metallurgical bond between the titanium coating and steel base, it is necessary to heat the base to the proper diffusion temperature during or immediately after deposition, and that it must also be cleaned prior to deposition to insure a non-porous metallic coating. The possible metallurgical explanation of the diffusion reaction is that when titanium is heated above

1616°F (880°C) an allotropic transformation occurs in which the close-packed hexagonal or "alpha" structure transforms into the body-centered cubic or "beta" structure, which is stable up to the meeting temperature (Figure 1). If the substrate temperature is below the transformation temperature, the "beta" structure transforms rapidly into the "alpha" structure; if, however, the substrate temperature is at the transformation temperature, the "beta" structure can be retained indefinitely. Hence, when titanium is deposited on an iron substrate which is heated above the titanium transformation temperature, the cubic "beta" titanium and cubic "alpha" iron structure readily form a solid solution diffusion zone on the surface of the substrate. An additional deposition of titanium metal on this zone produces a cohesive bond between the layer of titanium and the diffusion zone.

A metallographic examination of one good diffusion coating revealed a diffusion zone to be approximately 0.1 mil and the overlayer 1 mil thick. Nonporous diffusion coatings of titanium on steel panels have successfully withstood 200 hours exposure to salt spray and also typical mechanical deformation tests.

TEST MATERIAL AND EQUIPMENT

The titanium used in all evaporations was welding grade wire Ti75A. A spectographic analysis showed traces of manganese, iron, and tin. The commercial cold rolled titanium sheet was formed from Ti55A grade and contained traces of silicon, manganese, iron, copper and tin. The iron substrate 1 mil thick was cold rolled from 99.9% pure iron.

The titanium coatings were produced in a vacuum deposition apparatus constructed in the Physical Sciences Laboratory, U. S. Army Tank-Automotive Center. This apparatus was equipped with glass and aluminum bell jars. The vacuum system, which consisted of a mechanical oil pump in combination with an oil diffusion pump and a dry ice-trap, produced a vacuum of 10^{-4}mm Hg. Heavy duty transformers provided the current used in heating the substrates and evaporation filaments. Figure 2 is the vacuum apparatus used in evaporations. A detailed description of the vacuum system is given in Reference 1.

X-ray diffraction work was done with a G.E. XRD-3D diffractor equipped with a copper tube and a G.E. integrating pole figure goniometer (Figure 3), and a Norelco X-ray diffraction unit with an iron target. Figure 4 is a view of the pole figure goniometer without a sample in the specimen holder. The integrating feature of the goniometer is the reciprocating motion which allows the x-ray beam to cover a large number of crystals in the specimen. The pole densities at the various positions are achieved by the "alpha" and "beta" rotations (Figure 5). For every 360° change in "beta", or one complete revolution, the angle "alpha" changes in 5° increments. The x-ray transmission, or Schulz, technique is used between "alpha" = 0° to "alpha" = 55°. Figure 6 is the x-ray diffraction pattern of the transmission technique.

While the sample is in motion, the x-ray intensity is being recorded continuously on strip chart paper, and is marked at every change in "alpha" degrees. Figure 7 is a pole figure trace from "alpha" = 0° to "alpha" = 60°. Points of "n" times random intensity are marked off within every "alpha" change, and the corresponding "beta" angle is obtained. By plotting the "alpha" angle along the circumference on polar coordinate paper, a sterographic projection or a pole figure of the crystalline orientation of the metal is obtained. Therefore, by connecting all the points of constant intensity, a contour is obtained, which represents a two dimensional graphical visualization of a three dimensional crystalline orientation. Whenever possible the pole figure is plotted with respect to mechanical roll direction.

Commercially Rolled Titanium Foil

The commercial cold rolled titanium foil was prepared from sponge titanium produced by the Kroll (magnesium) process. Briefly, the 6000 pound ingot is double vacuum melted, and after conditioning, it is hot rolled to 0.150" between 1400°F - 1600°F. Then the material is strip rolled from 0.125" to 0.002" with intermediate anneals at 1250°F. The cold reduction is approximately 40% between annealing operations. The 0.002" strip is finally vacuum annealed at 1300°F and subsequently cold rolled to 0.0005" thickness without any intermediate annealing.

TEST PROCEDURE

Specimen Preparation

The vacuum deposition technique consisted of mounting the substrate approximately six inches above the evaporating source, shielding it during initial fusion and then evaporating at a rapid rate. The arrangement of the evaporation source, shutter, thermocouple and steel specimen is shown in Figure 8. Steel blocks, $2\frac{1}{2}$"x1"x$\frac{1}{4}$", were cut from strip steel, surface ground and cleaned in an ultrasonic degreaser. The latter dimensions were chosen as the thickest specimen that could be heated without overheating the bell jar. Coating of the thick steel specimens was accomplished by using heavier copper electrodes and two heavy duty transformers connected in parallel. Carbon boats, $2\frac{1}{2}$"x$\frac{1}{2}$"x$\frac{1}{4}$", were cut and shaped from carbon sheet and vacuum degassed at 1500°F for 30 minutes. Less dense but purer carbon required only a five minute degassing period. The coatings on steel were made at two temperatures, 1750°F and 1550°F, after a preliminary helium-argon flush and a vacuum degass. The higher temperature produced a diffusion bond, the lower a non-diffusion mechanical bond. The uniform coating obtained by heating the material at the diffusion temperature is shown, in Figure 9, in contrast with the uneven coatings caused by excessive heating. The appearance of the carbon boats at various stages is shown in Figure 10.

When the steel base is heated excessively above the diffusion temperature, metal flow occurs on hot-spot areas, which cause a pile-up of the coating material. These pile-up areas, which have a hard and bright surface, can be eliminated by corner clamping in the electrode and temperature control. Temperatures were measured with a Leeds and Northrup and a Lewis potentiometers using a 40 gauge iron-constantan and 20 gauge chromel-alumel thermo-couple wire, respectively. Thickness of the foils from the mica and steel substrates ranged from less than 1 mil to 2 mils.

X-Ray Technique

The metal films were cut into $1\frac{1}{4}''$ diameter specimens and mounted in the sample holder of the integrating pole figure goniometer. The specimen was set in the reflection position, and scanned automatically from $2\theta = 30°$ to $2\theta = 70°$ (Figure 11) at a rate of 2° per minute. After identifying the important reflection peaks, the basal (002) plane was selected for all pole figure determinations. A detailed description of the x-ray techniques is given in Reference (4) and (5). Three prominent reflection planes of the hexagonal crystal, designated by their Miller indices, are illustrated in Figure 12. The pole figure of the basal plane of mica was determined for comparison purposes, while the pole figures of the important planes of iron were assumed to be the same as reported in Barrett.

RESULTS AND DISCUSSION

The condensation of metal vapors on another metal surface in a high vacuum can produce either a mechanical or diffusion type of bonding, depending upon the temperature of the substrate. Below the diffusion temperature, the metal vapors condense on the surface without forming a metallurgical bond with the base metal. At or above the diffusion temperature, however, alloying occurs at the substrate surface, which produces a metallurgical bond between the overlayer and the base metal. Since corrosion resistance provided by both types of coatings is dependent on the quality of the coating, it is necessary the substrate be degassed and cleaned. Because of the nature of mechanical bonding, these surface treatments are much more important than in a diffusion coating. The importance of a clean surface for mechanical bonding was noticed when this type of coating failed after a few hours in the salt spray. An examination of the corrosion pattern revealed the failures started through pinhole areas, which enlarged into a pitted area and eventually undercut the coating.

The x-ray diffraction pattern of the titanium coating performed below the diffusion temperature (Figure 13) shows a slightly preferred orientation of the (011) pyramidal plane, while the reflection from the (010) prismatic plane, although much weaker has a noticeable preferred orientation. In contrast, the diffraction pattern of titanium deposited above the diffusion temperature (Figure 14) shows a weak, though slightly preferred, reflection from the (011) plane and a medium intensity reflection from the (002) basal plane.

The diffraction pattern (Figure 15) of the piled-up area consists of a broad, spotty line, which is characteristic of recrystallized metal. On the other hand, however, titanium evaporated on slightly heated cold rolled iron (Figure 16) shows a strong preferred orientation of the (002) basal plane. The pole figure of this layer (Figure 17), which was stripped from the iron base, consists of two double intensity regions in the direction of roll of the iron substrate. Although pole figures could not be plotted for titanium deposited at the elevated temperatures, the x-ray diffraction patterns, nevertheless, do illustrate the influence of temperature on the crystal orientations of the vacuum deposited coating.

Titanium evaporated on a non-metallic crystalline surface, such as mica, is characterized by crystal orientation that is quite different from the orientation produced on a metallic surface. The pole figure of a thin foil stripped from freshly cleaved mica surface (Figure 18) exhibits several large intensity regions which can be made symmetrical with respect to an arbitrary axis. This pole figure is noticeably different from the pole figure of basal plane of mica which consists of narrow regions radiating from the center. The diffraction pattern (Figure 19) shows, in addition to the basal and prismatic planes, a heavy line apparently due to the (110) plane of rutile (TiO_2).

Earlier work done on the texture of thin mechanically deformed titanium films has been reported by a number of authors[9,10]. These workers have investigated thin films that were carefully fabricated from small ingots under controlled laboratory conditions, and chemically etched to the final thickness. Using an x-ray film technique, Clark determined the basal plane texture of cold rolled titanium 4 mils thick. The noted feature of his pole figure is the double intensity band across the transverse with a maxima which occurs at 30° from the normal, which upon annealing dissociates into two separate region. Furthermore, investigators[11] have concluded that in cold rolling the (1010) direction of the hexagonal crystal is parallel to the roll direction, and that there is a tendency of the basal plane to rotate about this direction at 30° to the rolling plane.

The commercial titanium foil was commercially fabricated to a thickness of 0.5 mil from a 6000 lb. ingot extracted by the magnesium process. The pole figure reveals (See Figure 21) a more complicated structure. There are a series of structures located 70° from the normal in the roll direction. In addition, there are two intensity regions located in the roll direction and approximately 50° from the normal direction. The diffraction pattern of cold-rolled titanium (Figure 20) shows a fine grain structure with a strong reflection from the basal plane.

All of the titanium layers examined showed many of the characteristic lines of the titanium hexagonal crystal listed in Reference 6. Calculations of the "d" spacing of some important planes showed only a slight

change in the lattice constants. A comparison of the relative instensity of three crystallographic planes of various types of evaporations with a NBS power standard(7) is shown in Table 1. These intensities were determined with nickel filtered copper Kalpha radiation at 40 kilovolts-12 millamperes, using an ionization chamber operated at 1700 volts, and were roughly verified from diffraction patterns produced by iron Kalpha radiation.

Investigators have noticed that the oxidation rates of iron, nickel, and zinc crystals at high temperatures vary with the crystalline plane. Also, various crystal planes of the copper crystal have different corrosion rates in various acids. Even the physical properties such as shear or the electrical properties such as permeability are influenced, as in iron, by a preferred orientation. It must be remembered, however, that most of these studies were performed with single crystals, which can be readily produced in the laboratory. For practical applications, it is necessary, of course, to fabricate these crystals into a thin sheet or coating of polycrystalline metal. By mechanical cold rolling the metal or electrodeposition techniques, the preferred orientation of crystalline structures can be obtained. The problem is, however, more complicated during vacuum deposition, where a number of factors such as pressure, deposition rate, and temperature effect the crystal texture of the coating. Because of the nature of the vacuum deposition process, difficulties might be encountered in obtaining uniformly oriented metal coatings. Although the corrosion rates of the titanium hexagon crystal have not yet been investigated, its corrosion resistance to many atmospheres and liquid media are well known. Consequently, in view of the results of previous investigations, previous work has established that as long as the cohesive titanium coatings are free from any surface defects, it will provide excellent corrosion resistance, regardless of the nature of crystal orientation.

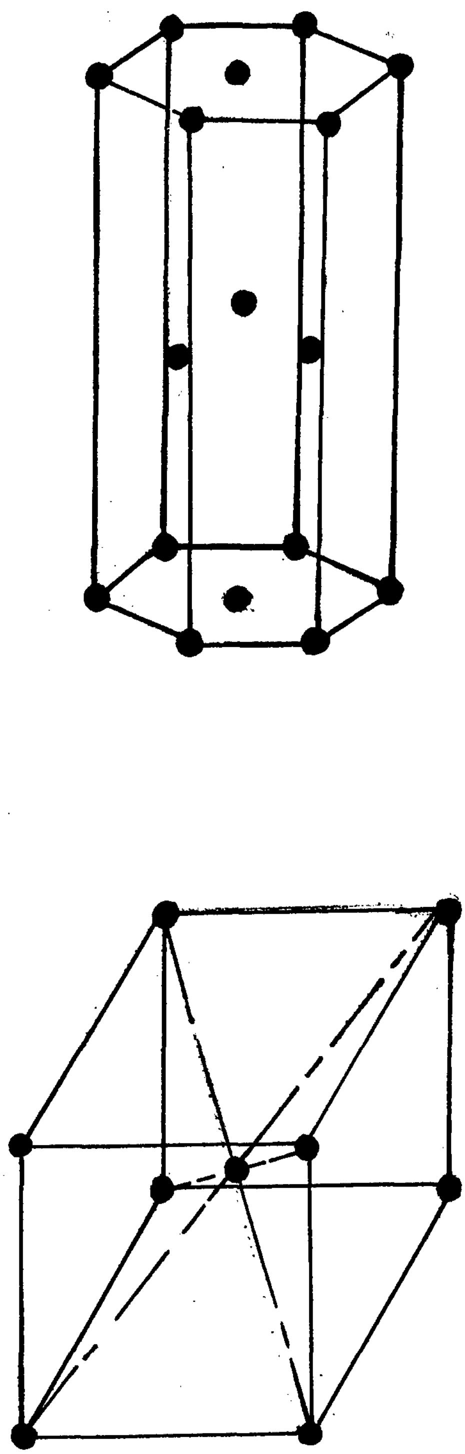
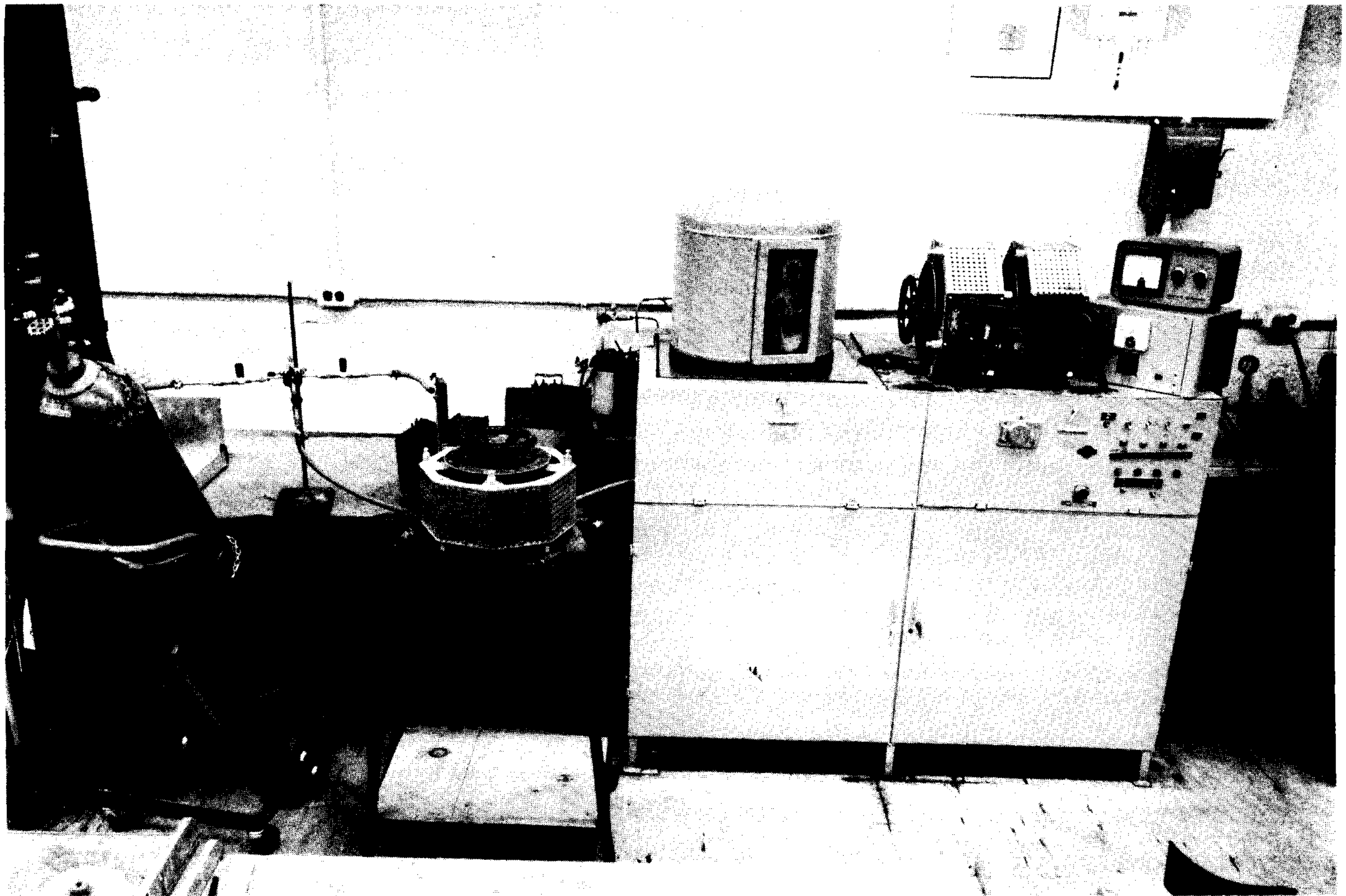
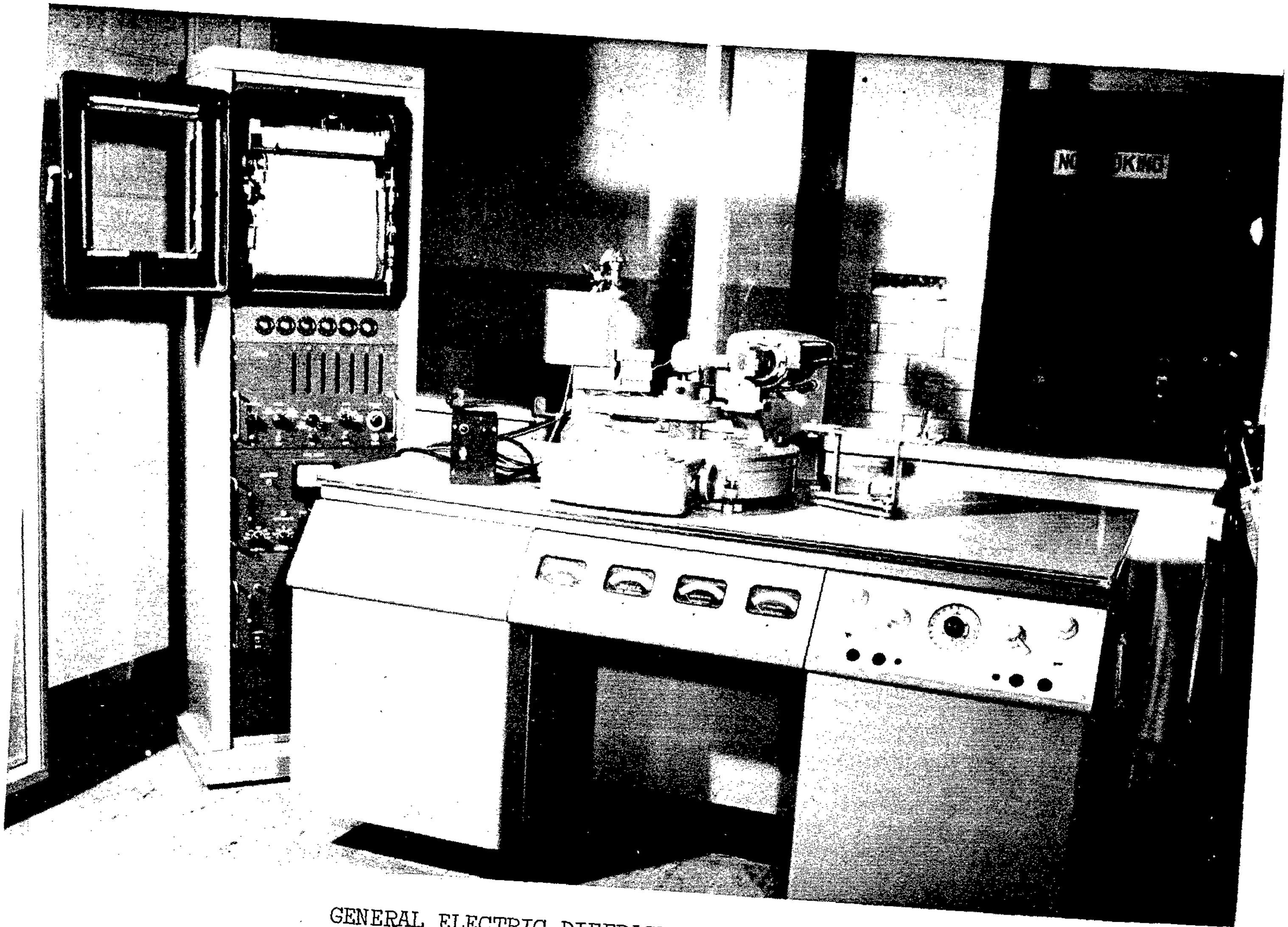


FIGURE 1



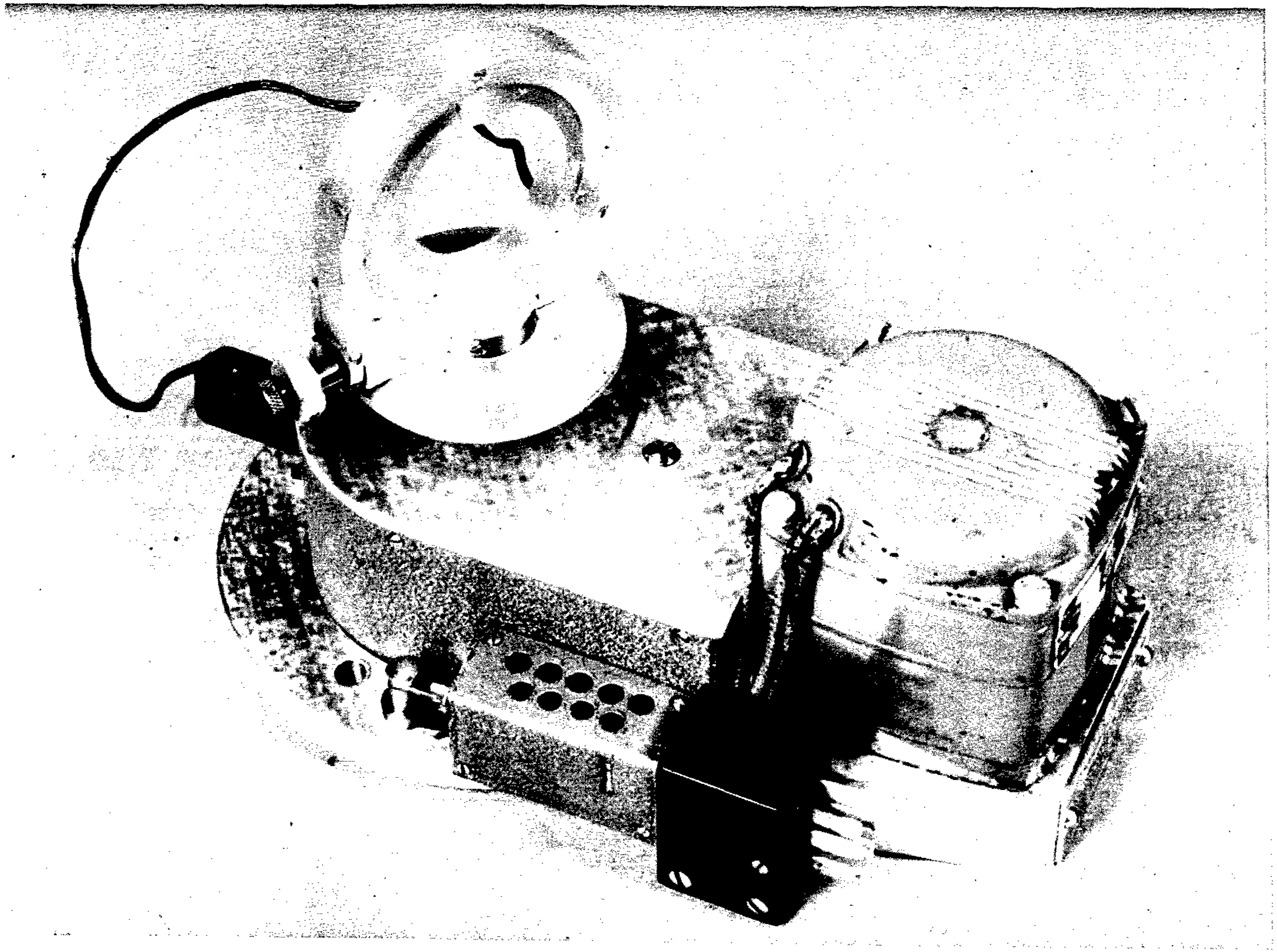
VACUUM DEPOSITION APPARATUS

FIGURE 2



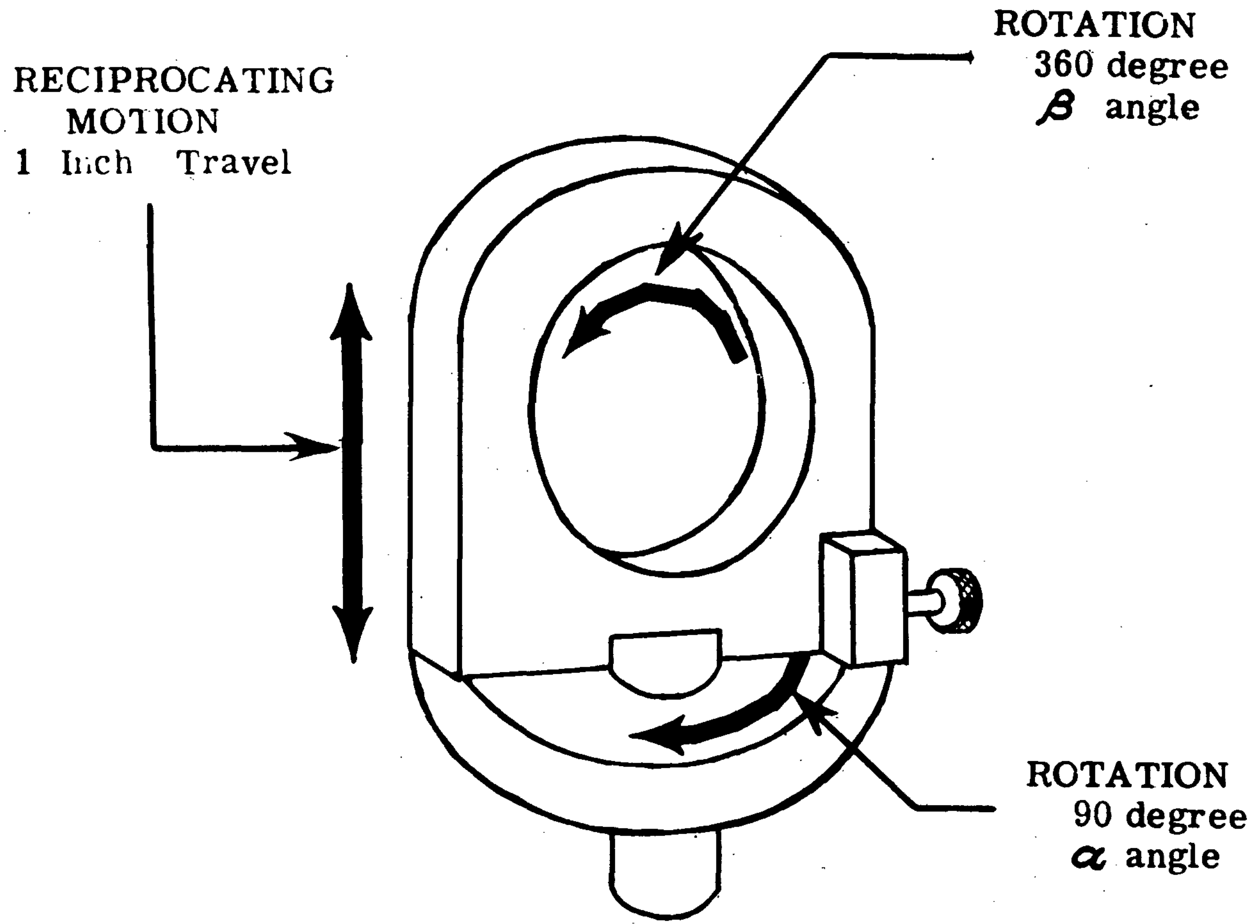
GENERAL ELECTRIC DIFFRACTION UNIT WITH GONIOMETER

FIGURE 3



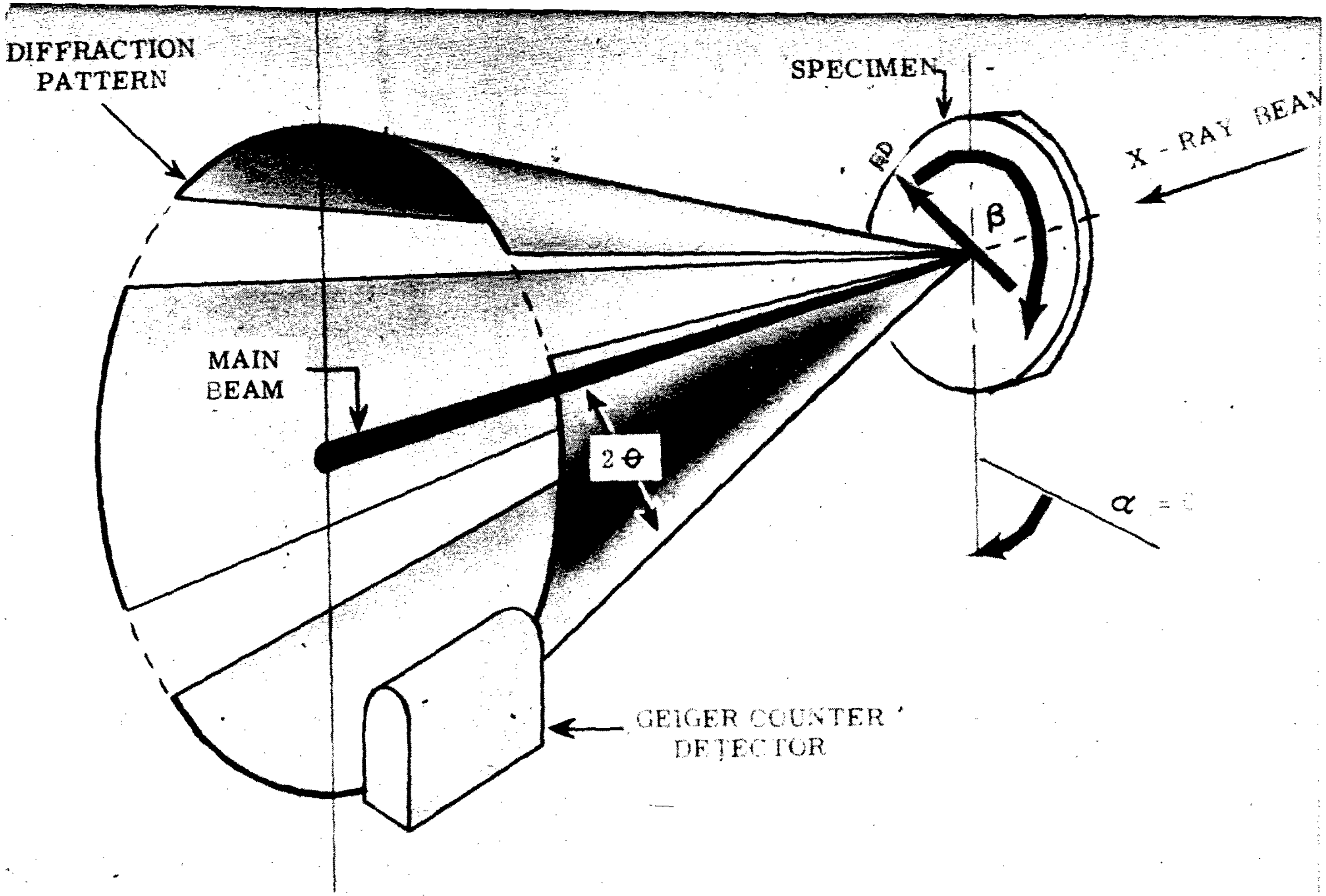
THE INTEGRATING POLE-FIGURE GONIOMETER

FIGURE 4



MOTIONS OF SPECIMEN IN THE
INTEGRATING POLE-FIGURE GONIOMETER

FIGURE 5



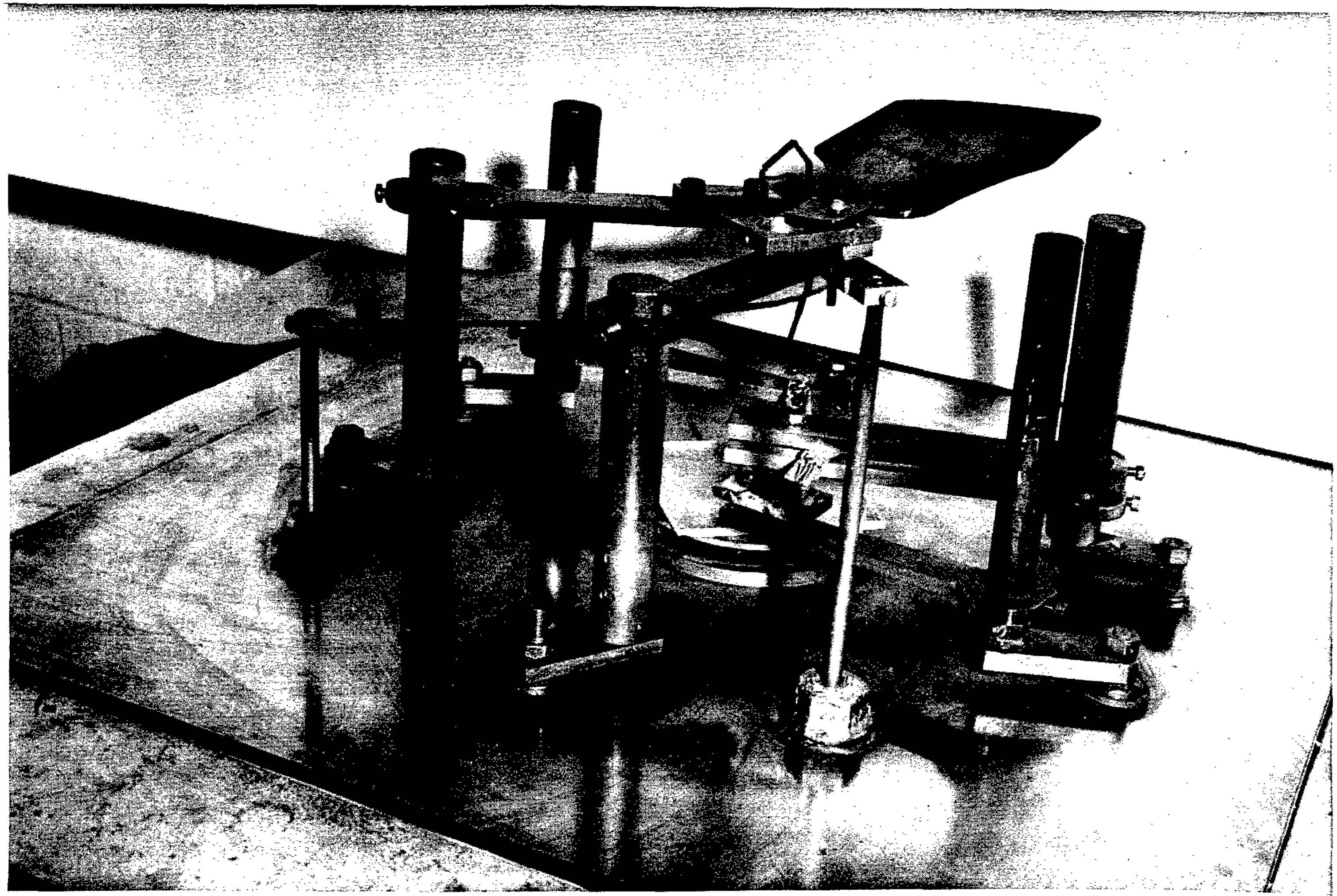
TRANSMISSION TECHNIQUE

X RAY DIFFRACTION PATTERN OF THE TRANSMITTED BEAM

FIGURE 6

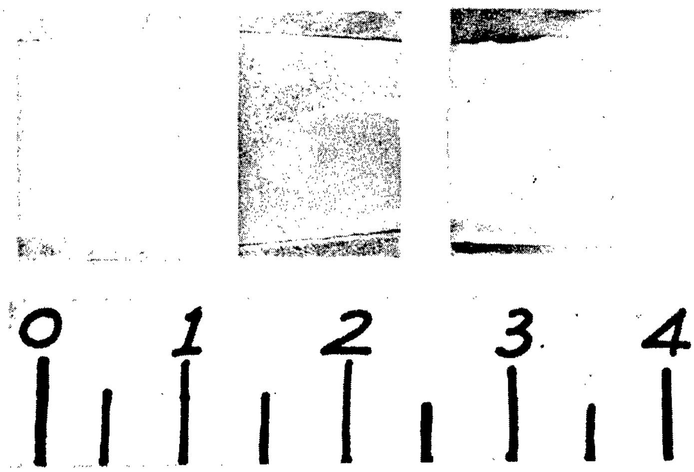
BETA ANGLE
POLE-FIGURE TRACE OF THE BASAL (002) PLANE OF COLD ROLLED TITANIUM

FIGURE 7



ELECTRODE ARRANGEMENT OF VACUUM SYSTEM

FIGURE 8



TITANIUM COATINGS ON THICK STEEL SPECIMENS

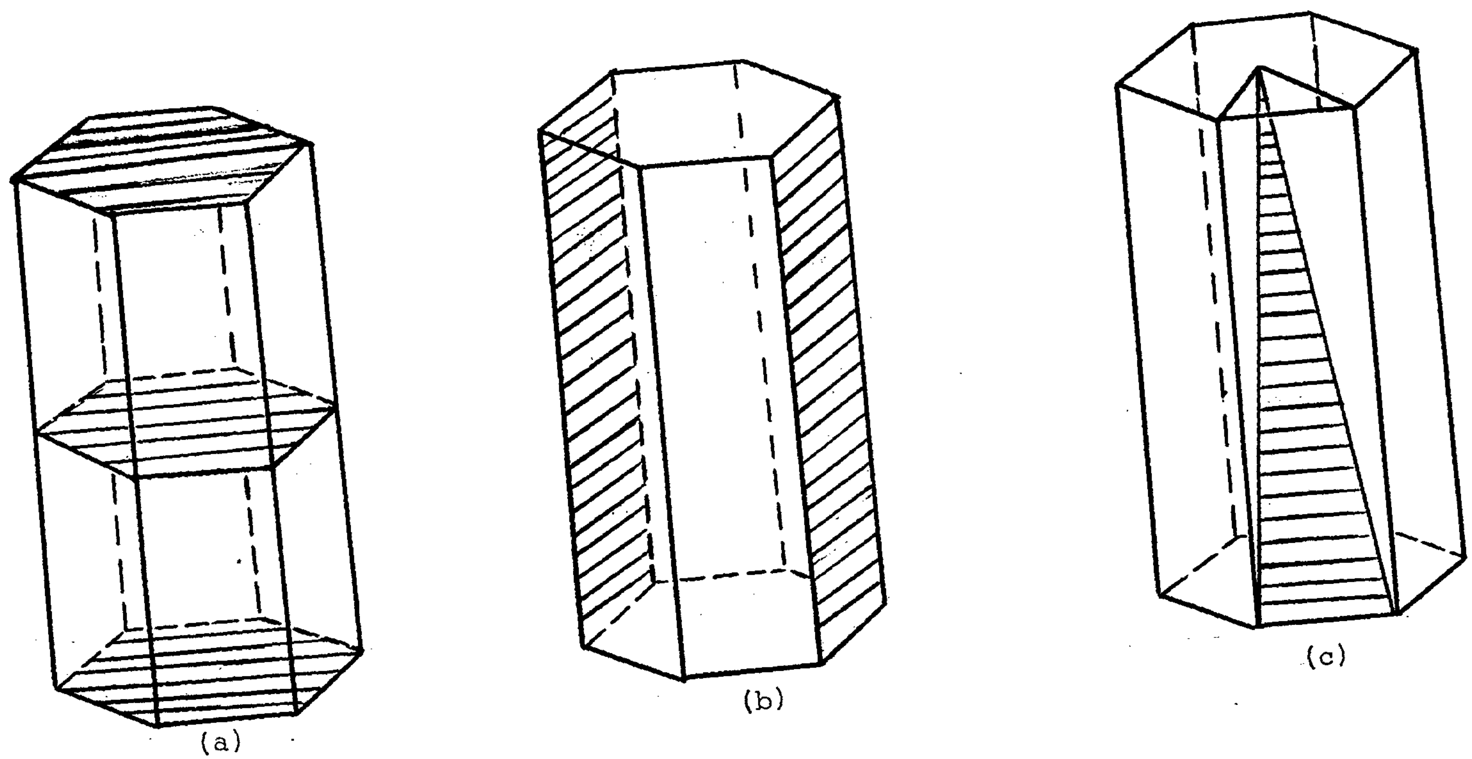
FIGURE 9

CARBON BOATS USED IN THE EVAPORATIONS

FIGURE 10

X-RAY SPECTRAL TRACE OF COLD ROLLED TITANIUM
FOIL WITH CRYSTAL PLANE DESIGNATIONS

FIGURE 11

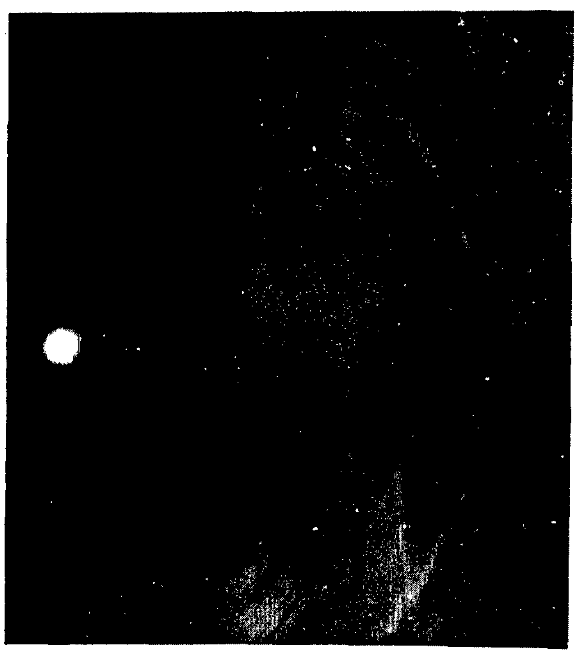


SOME IMPORTANT CRYSTAL PLANES OF TITANIUM (LINED AREAS)
(a) BASAL (002): (b) PRISMATIC (010); (c) PYRAMIDAL (011)

FIGURE 12

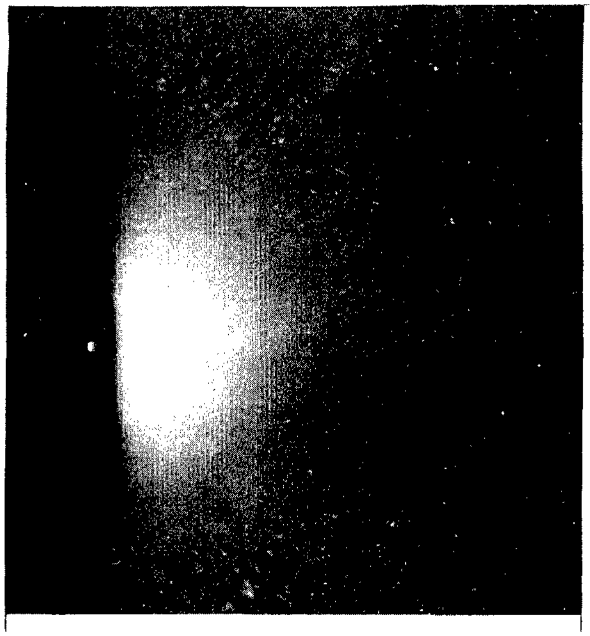
DIFFRACTION PATTERN OF TITANIUM
DEPOSITED ON STEEL HEATED AT 1500°F

FIGURE 13



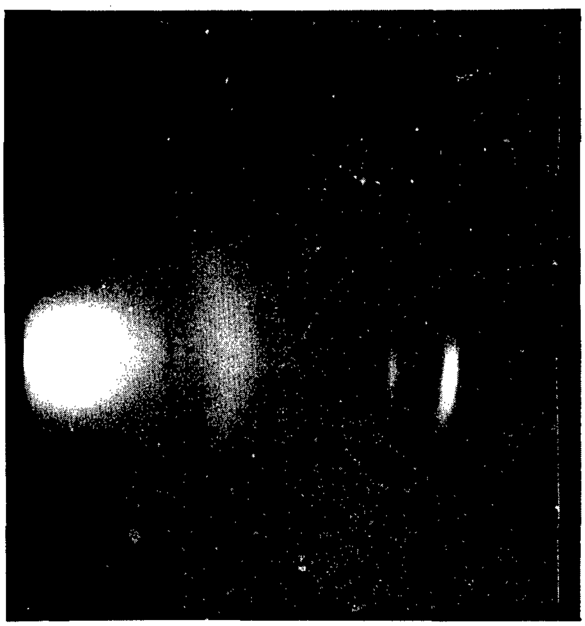
DIFFRACTION PATTERN OF TITANIUM
DEPOSITED ON STEEL SLIGHTLY ABOVE 1750°F

FIGURE 14



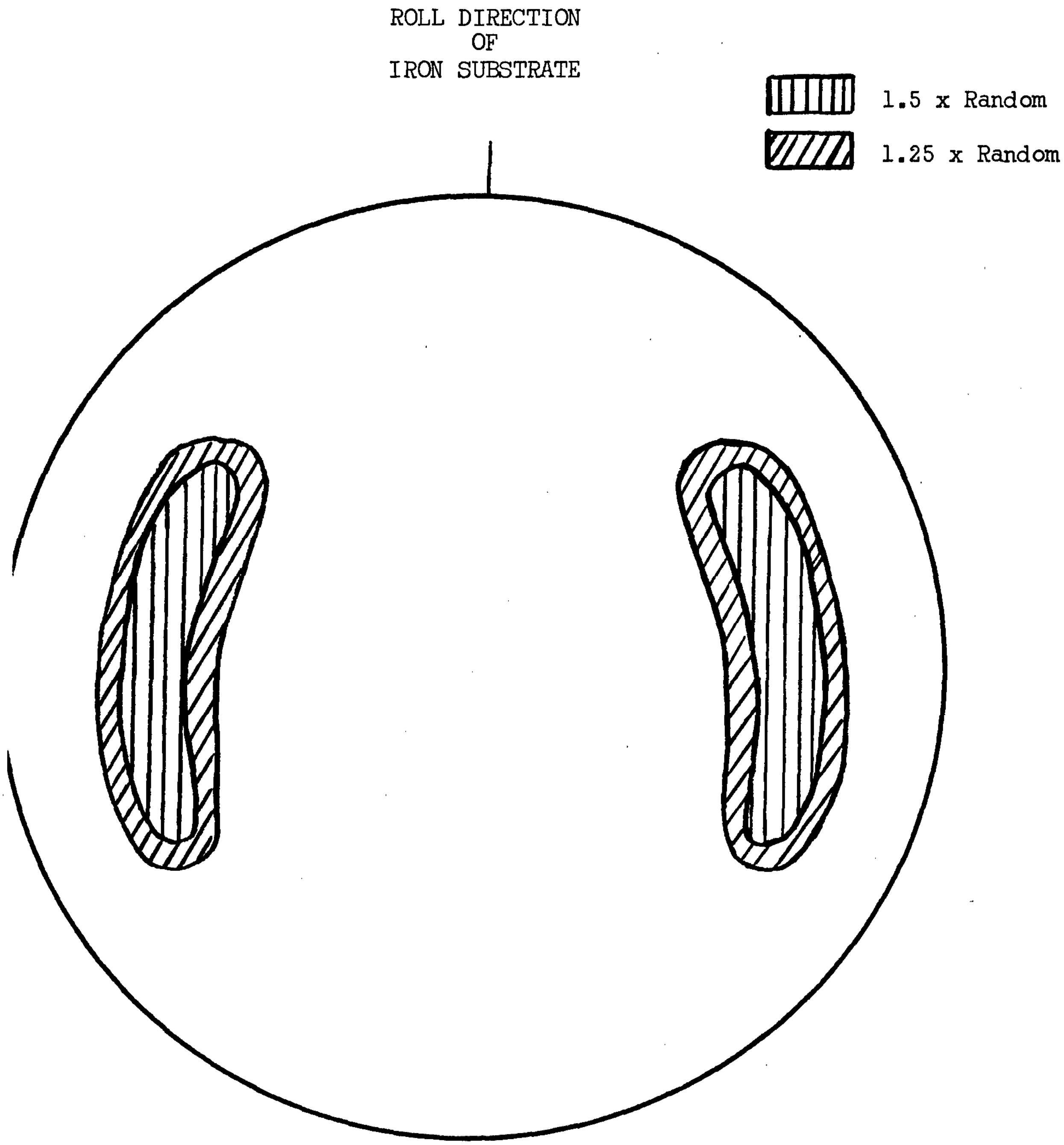
THE DIFFRACTION PATTERN OF THE
PILED-UP TITANIUM AREA

FIGURE 15



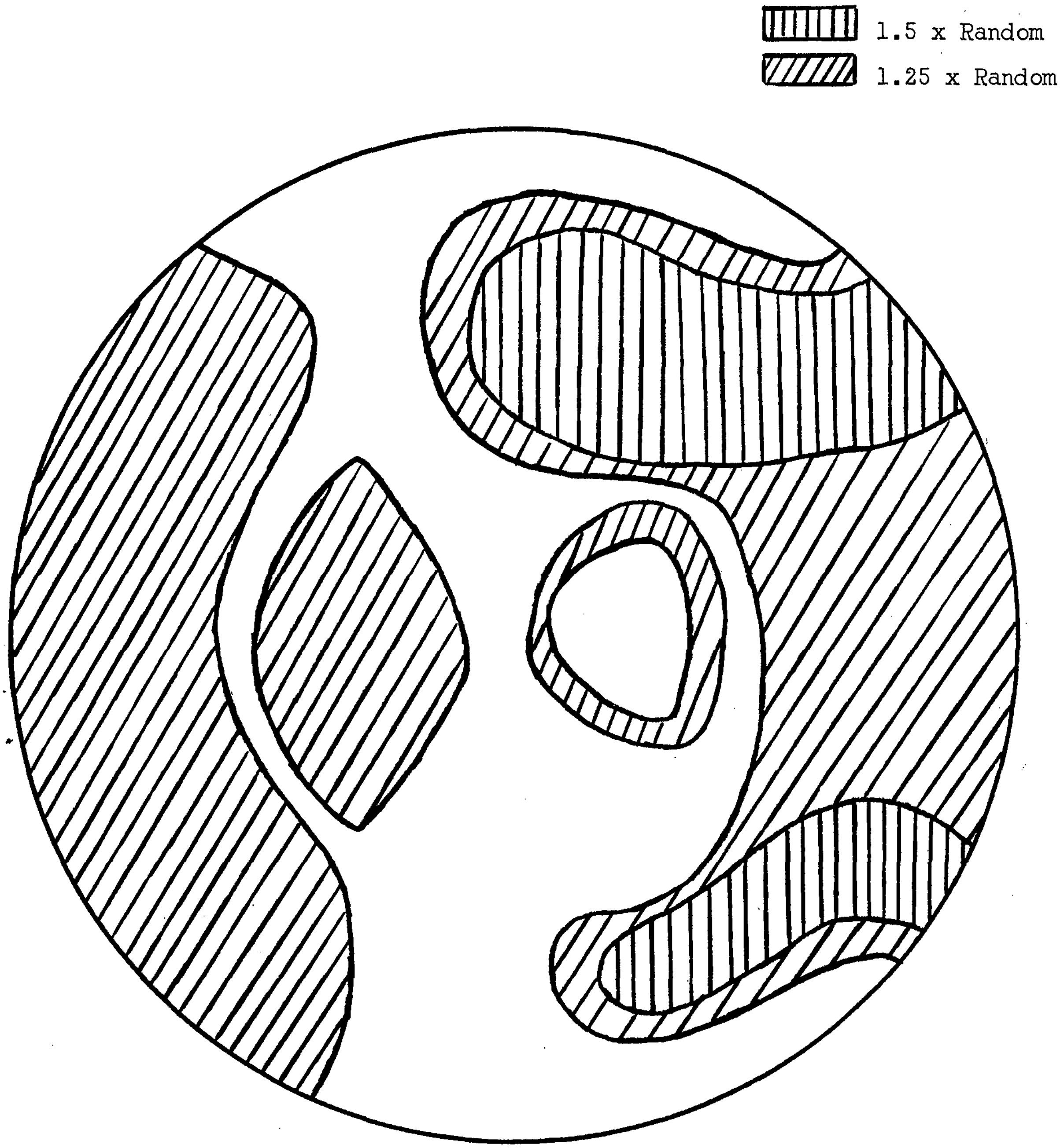
DIFFRACTION PATTERN OF TITANIUM
DEPOSITED ON A SLIGHTLY HEATED IRON SURFACE

FIGURE 16



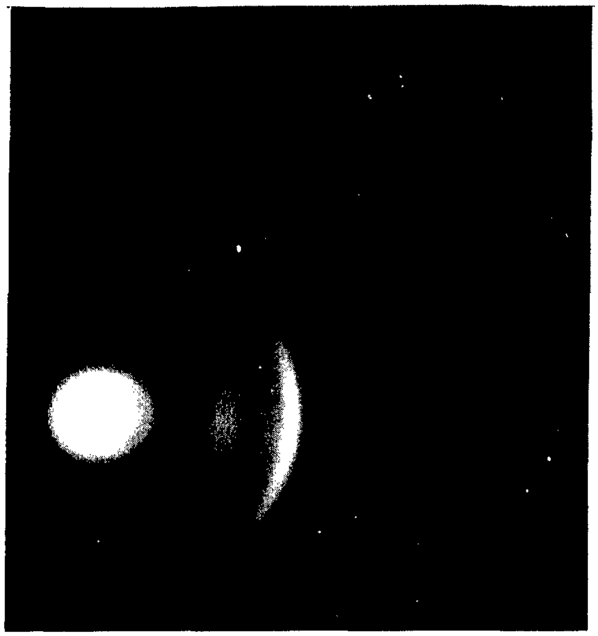
BASAL POLE-FIGURE ON TITANIUM EVAPORATED
ON COLD ROLLED IRON

FIGURE 17



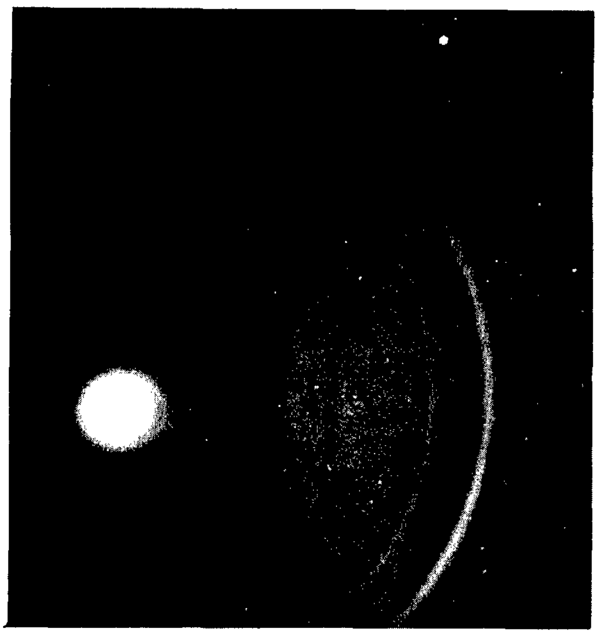
BASAL POLE-FIGURE OF TITANIUM
EVAPORATED ON MICA

FIGURE 18



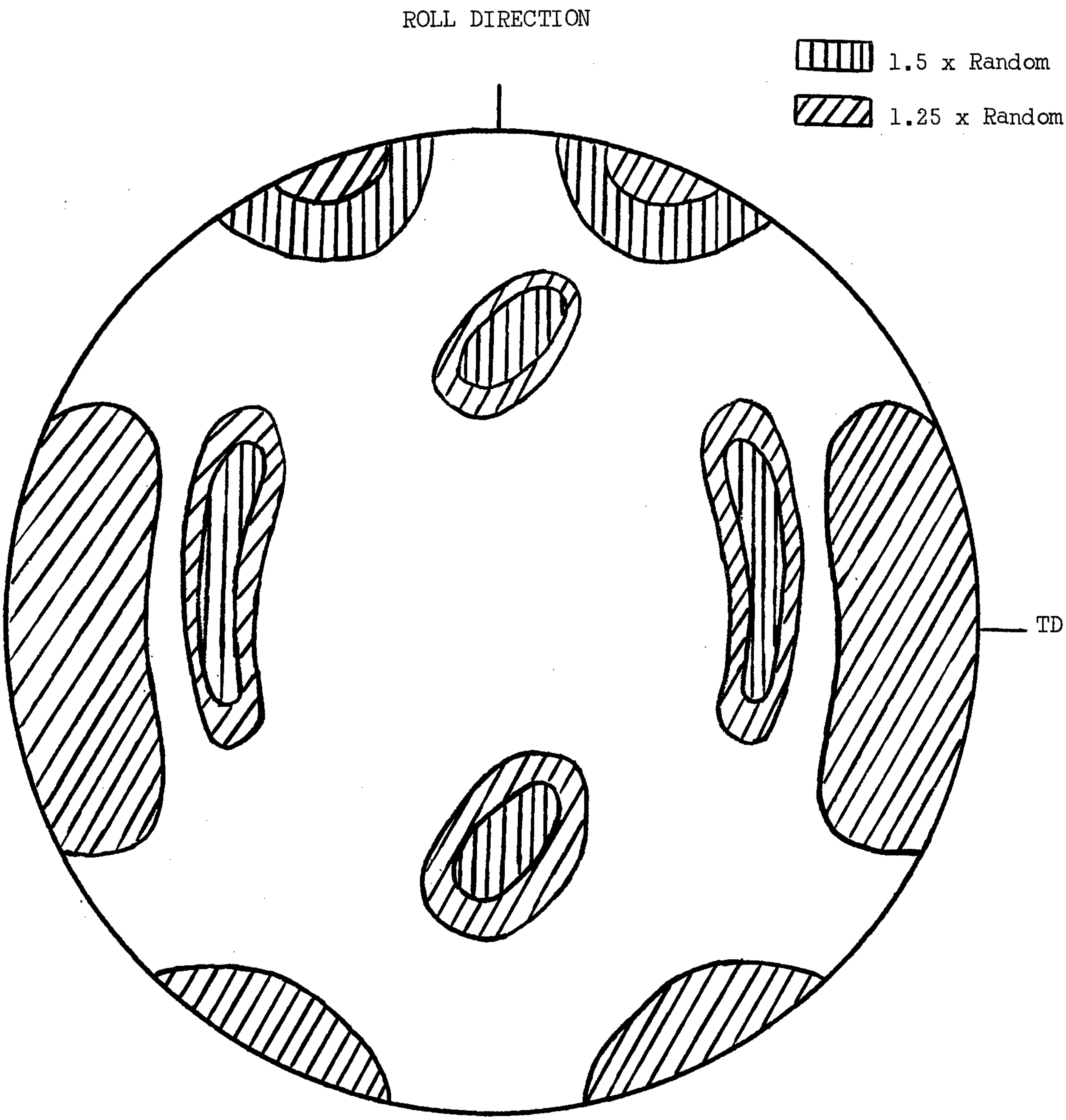
DIFFRACTION PATTERN OF TITANIUM EVAPORATED ON MICA

FIGURE 19



DIFFRACTION PATTERN OF COLD ROLLED TITANIUM RIGHT TO LEFT:
(011) PYRAMIDAL, (002) BASAL; AND (010) PRISMATIC LINES

FIGURE 20



BASAL POLE-FIGURE OF COMMERCIAL COLD ROLLED TITANIUM

FIGURE 21

TYPE OF TITANIUM	1	2	3
FOIL (COLD ROLLED)	002	012	011
ON DIFFUSED IRON	002	012	103
FROM DEGASSED COLD IRON	002	011	012
FROM UNDEGASSED COLD IRON	010	---	---
FROM MICA	103	010	002
NBS POWDER STANDARD	011	010	002
ON STEEL BELOW 1616°F	011	002	---

RELATIVE INTENSITY OF SEVERAL PROMINENT REFLECTION PLANES

TABLE 1

APPENDIX

FIGURES AND TABLES

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Geiger counter and x-ray film techniques were used to study the crystalline structures of thin titanium films and coatings formed by vacuum deposition. An x-ray analysis of the various titanium films was used to determine the effects of temperature and base metal orientation upon the structures of the deposited films. Results indicate that the degree of preferred orientation of the films can be altered by variations in the vacuum deposition technique. Attempts were made to correlate film orientation with adhesion and corrosion resistance properties.

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